

STAGE REFERENCE MANUAL

(Geology Version)



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OPTICAL INFORMATION

It cannot be stressed enough, how important it is to have the microscope set up correctly, especially when working at high magnifications or when looking at difficult objects such as small fluid inclusions. The following few paragraphs detail the setting up of the most important features of the microscope such as the light source and condenser lens.

To help explain the setting up procedures for the microscope, a simple line drawing showing the essential parts of the microscope's optical path is shown opposite.

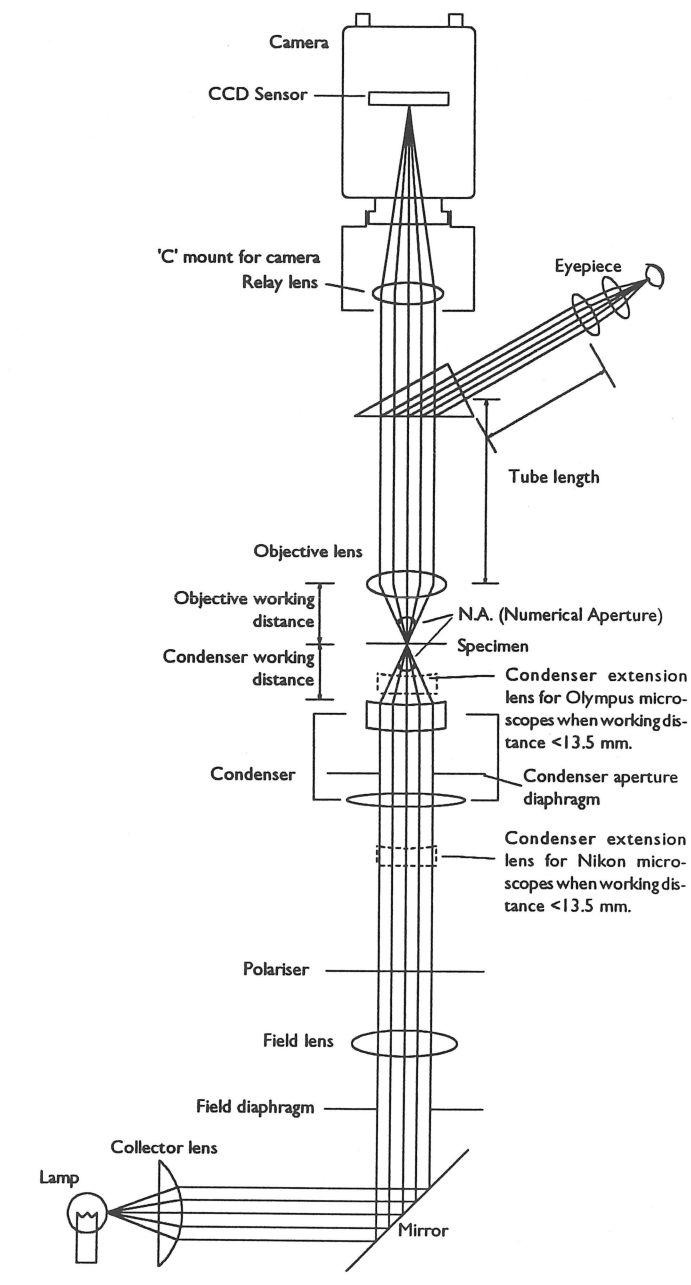
If the condenser working distance is less than 13.5 mm, we can supply a condenser extension lens for both the Olympus and Nikon microscopes under part numbers CEO and CEN respectively (see drawing). For other microscopes please contact us.

NOTE:-If the stage clamps are used with the Olympus microscope the CEO lens is NOT required.

Light source centring and focus

The adjustment of the light source is only necessary when the microscope is first installed or when the bulb is replaced, provided that the adjustment's made are not disturbed.

- 1) Open the field diaphragm on the base of the microscope to it's largest extent and place a flat, thin piece of paper over it.
- 2) Turn the light source to maximum output.
- 3) Slide the lamp housing backwards and forwards until the image of the bulb filament is visible on the paper.
- 4) To centre the filament, adjust both the lamp vertical centring ring and the lateral centring screw on the lamp housing.
- 5) Pull the lamp housing slightly forward until the filament image is diffused, or alternatively fit a diffusing filter.



Centring the condenser lens

It is extremely important to have a focused, centred and condensed light beam at all times and to ensure that the field diaphragm and condenser aperture diaphragm are set correctly.



1) Select the 10X lens and close the field diaphragm on the microscope base to it's smallest size. Rotate the condenser focus knob, moving the condenser up and down until a sharp image of the field diaphragm is seen clearly in the eyepiece.

2) Using the two condenser centring screws bring the field diaphragm image into the centre of the field of view.

3) Adjust the field diaphragm so that the image of the diaphragm is about the same as that of the field of view. If it is still not centred use the condenser centring screws again.

Use of field diaphragm

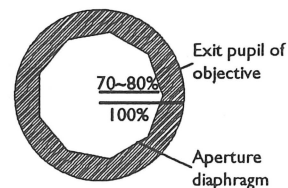
The field diaphragm controls the diameter of the illuminated area on the specimen surface in relation to the field of view of the microscope. By stopping down the field diaphragm until it is slightly larger than the field of view, it can reduce stray light, which in turn increases image definition and contrast.

Use of condenser aperture diaphragm

This adjusts the numerical aperture (N.A) of the illuminating system of the microscope and determines the resolution, contrast and depth of focus.

In general it should be stopped down to 70~80% of the numerical aperture of the objective lens for best overall results.

After completing focus adjustment, remove one of the eyepieces and look into the empty eyepiece tube. Adjust the size of the diaphragm, observing the image of the diaphragm which is visible on the bright circle of the objective exit pupil.



Heat filter

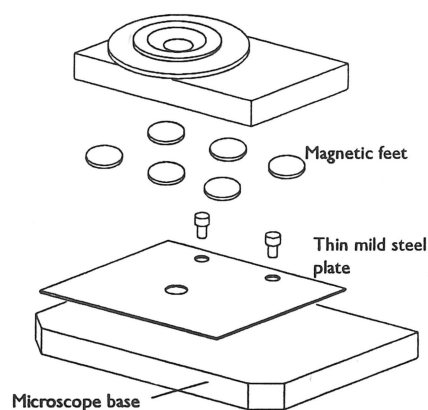
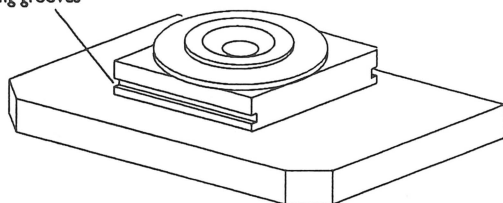
An infra-red heat filter should be placed in the path from the light source as this can effect the specimens's temperature due to absorption of heat. This becomes more noticable when lamp bulbs of 100 watts are used.

A suitable filter is available for both the Nikon and Olympus microscope, part number MBN11500.

MICROSCOPE MOUNTING

Six self adhesive magnetic feet are supplied which can be used together with a thin mild steel plate, to attach the hot stage to the microscope's mechanical base. The HFSG91 can also be mounted using the clamping grooves on the side of the body. If it is not practical to use the magnetic feet, double-sided adhesive tape can be used.

HFSG91 clamping grooves



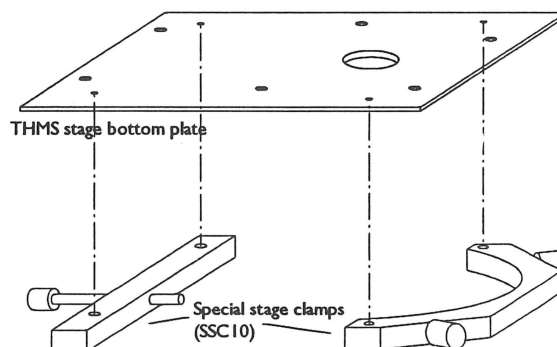
For very high magnification and stability, especially when using the CS196 Cooling unit, the microscope's X,Y table can be removed and the THMS stage fitted directly to the microscopes's circular sub stage.

MOUNTING ARRANGEMENT FOR NIKON AND OLYMPUS MICROSCOPES

This arrangement uses a set of SSC10 stage clamps which are simply screwed to the bottom of the existing THMS body using four cap screws which are provided. Some of the older THMS stages will not have the correctly drilled bottom plate and will have to be modified. Contact us for details.

The three positioning screws are used to centre the silver block window as follows.

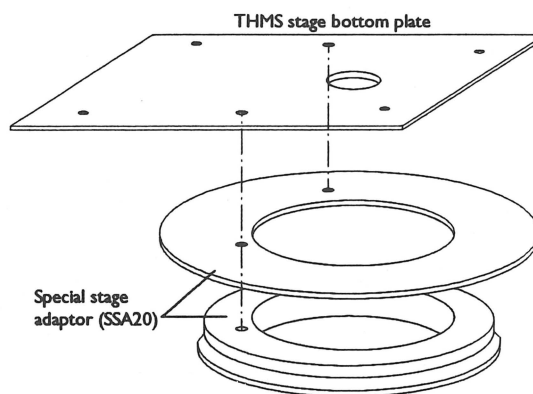
- 1) Loosen the three screws and position the stage and clamps on the microscope's sub stage.
- 2) Select a 10X lens and focus on the window.
- 3) Using the long screw, adjust the position of the stage in the X direction until it is centred.
- 4) Gradually adjust and tighten the two smaller screws in order to adjust the stage in the Z direction.



MOUNTING ARRANGEMENT FOR ZEISS AXIOPLAN AND AXIOPHOT MICROSCOPES

For the Zeiss Axioplan and Axiophot microscope, a special adaptor is available which is screwed to the bottom of the THMS body using the two 12mm screws provided. These replace the two 6mm screws already used in the base plate, as shown in the diagram.

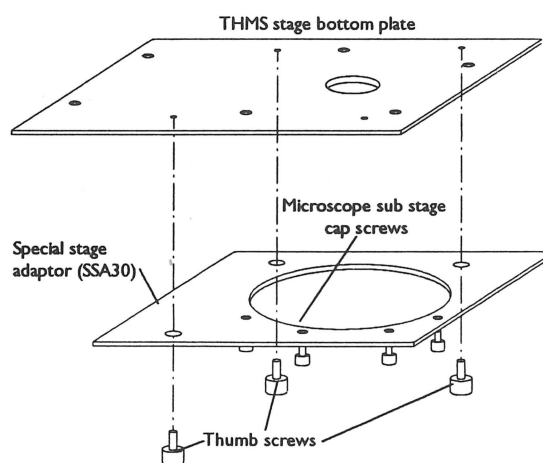
In order to centre the heater block and window, adjust the microscope's spring loaded screws which hold the SSA20 in place. If this is not enough adjustment, the SSA20 adaptor can be moved slightly by loosening the mounting screws and moving the stage body before re-tightening them again.



MOUNTING ARRANGEMENT FOR ZEISS AXIOSKOP MICROSCOPES

For the Zeiss Axioskop a special adaptor is available which is screwed to the bottom of the THMS body using the three thumb screws provided (see diagram). As these screws are also used for centring the heater block and window they should not be tightened until the complete assembly is on the sub-stage. Fit as follows:

- 1) Screw the SSA30 to the THMS body using the screws provided until they are finger tight.
- 2) Mount the complete assembly to the Zeiss sub-stage using the four screws already in the Zeiss sub-stage.
- 3) Select a 10X lens and centre the heater block and window by moving the THMS body. Tighten the mounting screws.



OBJECTIVE LENSES

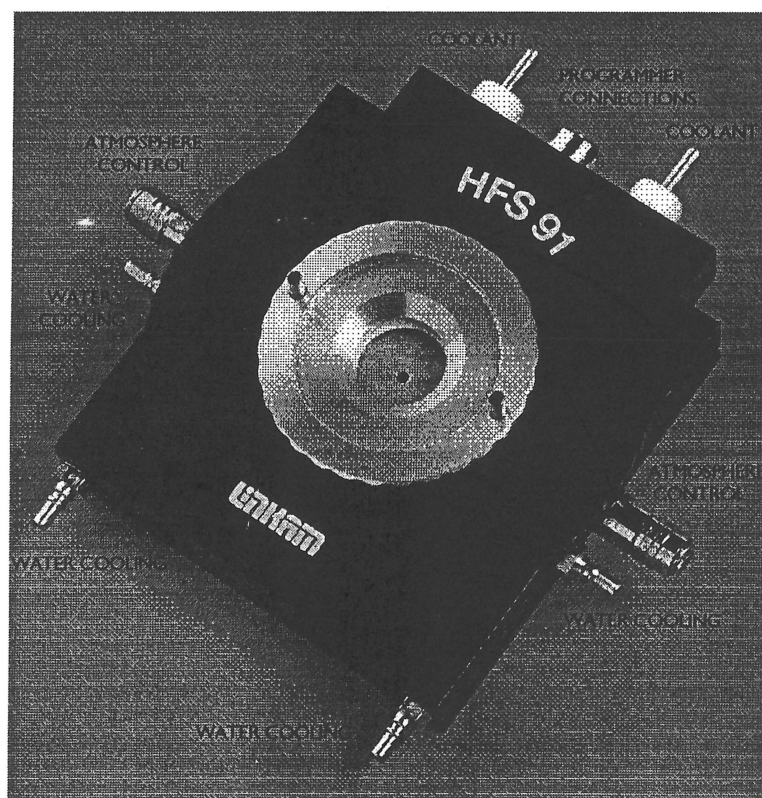
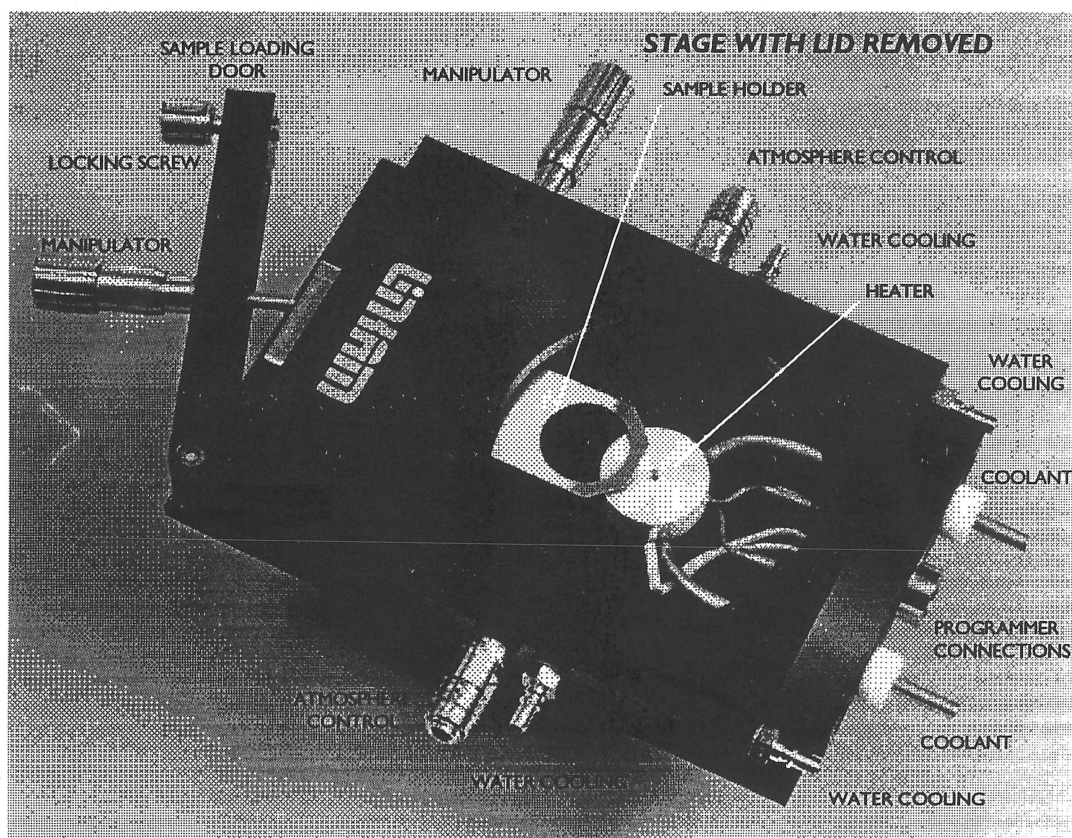
For use at high magnifications special lenses are required which have long working distances, a selection of which are shown below. Some of the Olympus lenses may be used on the Nikon microscope but because of tube length differences the magnification may not be exact. This is also the case if Nikon lenses are used on Leitz microscopes.

For correct lens magnification the lens must be matched to the tube length of the microscope (see optical drawing).

Over the page there is a list of lenses suitable for use with the heating/freezing stage.

OBJECTIVE LENS		NUMERICAL APERTURE	WORKING DIST (mm)	SPECIAL LID
OLYMPUS				
MS plan achromat MSPL10X				
Ultra long working distance ULWMSPL20X		0.4	11.0	
Ultra long working distance ULWMSPL50X		0.55	8.0	
** Excellent lens which also works well on the NIKON **				
Ultra long working distance MS plan achromat 80X				Yes.
NIKON				
MSK 00101CF E PLN LWD 10X . (cover glass corrected)				
MSJ 05200 CF N PLN ELWD 20X (cover glass corrected)				
MSJ 05400 CF N PLN ELWD 40X (cover glass corrected). Recommended for stage use		0.55		
MTJ 60100 CF M PLN ACH 10X	May require 160 to 210 mm tube length adaptor if the microscope is not a metallurgical type	0.25		
MTJ 63200 CF ELWD M PLN ACH 20X				
MTJ 63400 CF ELWD M PLN ACH 40X		0.5	10.1	
MTJ 63600 CF ELWD M PLN ACH 60X			4.90	Yes.
MTJ 67900 CF ELWD M PLN ACH 100X	Recommended for stage use	0.75	4.7	Yes.
LEICA (LEITZ)				
559121	L10/0.22P Long working distance 10 times polarised	0.22	15.8	
559122	L20/0.32P Long working distance 20 times polarised	0.32	6.73	
559123	L25/0.22P Long working distance 25 times polarised	0.22	14.7	
559124	L32/0.40P Long working distance 32 times polarised	0.40	6.55	
519760	EF10/0.25 Long working distance 10 times	0.25	6.78	
519434	EFL20/0.32 Long working distance 20 times	0.32	6.73	
519679	LL25/0.35 Long working distance 25 times	0.35	13.7-14.3	
519435	L32/0.40 Long working distance 32 times	0.40	6.55	
518087	EF10X Phase Contrast .. 1A	0.25	6.78	
518076	EFL20X Phase Contrast .. 1A	0.32	6.73	
518077	L32X Phase Contrast .. 1A	0.40	6.55	
569231	NPL FL 10/0.22 Infinity corrected	0.22	11.8	
569216	LL 20/0.40 Infinity corrected	0.40	10.1	
569244	PLAN L25/0.40 Infinity corrected	0.40	11.0	
569247	PLAN L50/0.60 Infinity corrected	0.60	6.8	
569107	H 20/0.40. For high temp TH1500.Corrected for 1.8 mm quartz.	0.40	8.2	
569109	H 32/0.60. For high temp TH1500.Corrected for 1.8 mm quartz.	0.60	5.8	
567002	PLANH 20/0.40. For high temp TH1500.Corrected for 1.8 mm quartz.	0.40	12.6	
567003	PLANH 40/0.60. For high temp TH1500.Corrected for 1.8 mm quartz.	0.60	7.1	
567000	PLAN L20X		11.0	
567004	PLAN L40X		6.8	
ZEISS JENA				
302358:372.26 .. Planachr. LD 8*/0.1 pol. (cover glass corrected)		0.1	32.7-37.7	
302357:372.26 .. Planachr. LD 16*/0.2 pol (cover glass corrected)		0.2	14.5-17.5	
302349:371.26 .. Planachr. LD 25*/0.5 pol (cover glass corrected)		0.5	11-13.5	
302355:002 K 40X (cover glass corrected)		0.5	17-18	
CARL ZEISS				
440120	Non flat Achrostigmat 5X (cover glass corrected)	0.12	11.0	
440130	Non flat Achrostigmat 10X (cover glass corrected)	0.25	6.5	
440020	Flatfield Achroplan 4X ... (cover glass corrected)	0.1	11.5	
440320	Flatfield Plan Neofluar5X (cover glass corrected)	0.15	13.6	
440330	Flatfield Plan Neofluar10X (cover glass corrected)	0.30	5.6	
440844	Flatfield Achroplan LD20X (cover glass corrected)	0.4	10.9	
440850	Flatfield Achroplan LD32X (cover glass corrected)	0.4	6.9	
442840	Flatfield Epiplan LD 20X	0.4		
442850	Flatfield Epiplan LD 50X	0.5	7.0	
CONDENSER LENSES				
OLYMPUS				
BH2-LWCD	Long working distance condenser	Use LINKAM lens CEO		
NIKON				
MBL 16100	Long working distance Achromat condenser	Use LINKAM lens CEN		
LEICA (LEITZ)				
513501		4.0	0.7	
513502		15	0.55	
513538		30	0.35	
ZEISS JENA				
304314:024.24 .. LD-Condenser ... 0.5 pol				
HEAT ABSORBING FILTER				
MBN1150 45 mm heat absorbing filter for Nikon and Olympus microscopes				

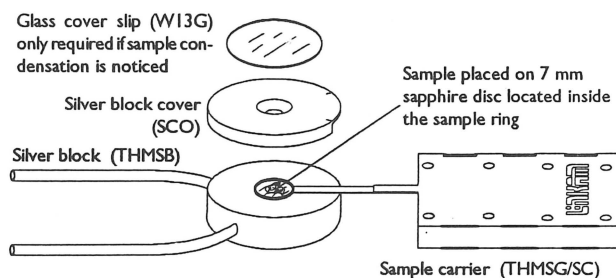
THMSG600/HFSG91 STAGE CONNECTIONS



THMSG 600 SAMPLE PREPARATION AND ACCESSORIES

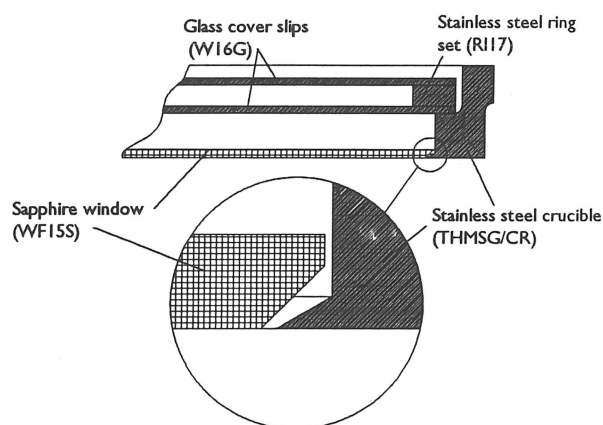
When working with fluid inclusions both the optical performance and the temperature performance becomes critical. It is very important to avoid the use of too many glass cover slips as each layer effects the sharpness of the image seen by the microscope. The arrangement shown here uses only one glass cover slip over the silver lid and one in the top lid assembly as shown on the next page. However, if there is no condensation on the sample, the cover slip over the silver lid can be removed.

To obtain a very even temperature around the sample, a cover is placed over the silver block to form a miniature 'furnace'. The cover has a small slot in the side to allow the sample carrier to move. With this arrangement the stage and programmer can achieve an accuracy of better than 0.2°C.



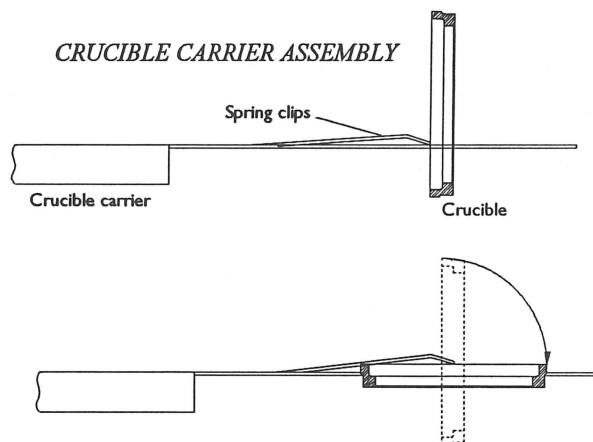
An alternative arrangement shown here allows the sample to be loaded from the THMS side door without removing the lid. It has a sapphire window which is placed inside a stainless steel crucible. The edge of the window and the crucible are bevelled so that the window cannot fall out as the assembly is inserted or removed through the side door.

CROSS SECTION OF CRUCIBLE AND WINDOW



The crucible is held in place in the carrier by two spring clips as shown in the assembly drawing opposite. The crucible should slide under the two spring clips as it is turned through 90° so that the spring clips rest on the crucible's rim. The assembly should then be placed on a clean flat surface to allow the window to be fitted. Take care that the window is not 'tilted' as this will cause an air gap when placed on the silver block. The Linkam Sample Loading Station is ideal for this purpose.

CRUCIBLE CARRIER ASSEMBLY



For accurate results it is most important that the surface of the block, window and carrier are extremely clean as any particles of dust, grease or skin will cause air gaps between the block and the window resulting in temperature errors. Ideally the windows should always be handled using the Linkam vacuum tweezers.

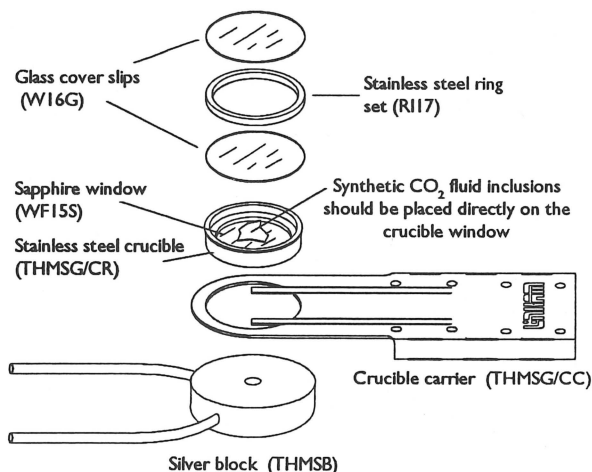
For precision work the stainless steel rings and cover slips can be used to form a 'double glazed' environment.

When working with a synthetic fluid inclusion (e.g. CO₂) it should be placed directly on the crucible's window.

The crucible has the advantage over the silver lid assembly shown above, in that it is easier and quicker to use when looking at a large number of samples.

Using this crucible arrangement, can however, produce a very small error which can be calibrated out using the simple built-in sample calibration feature of the temperature programmer (see SAMPLE CALIBRATION FOR THE THM600/HFS91 STAGES in the programmer's reference manual).

Before changing the sample calibration of the programmer the follow-



ing should be checked.

- 1) The silver block should be clean and flat so that the crucible 'sits' correctly on the block without any air gaps.
- 2) Make sure that the sapphire window in the silver block is level with the silver block top surface as this would cause the crucible to 'tilt' to a different position each time it is used and may cause the temperature values to be un-reproducible.

NOTE: Once the crucible arrangement has been used and if the programmer's sample calibration has been changed, the user must reset the sample calibration in the programmer before using the silver lid arrangement.

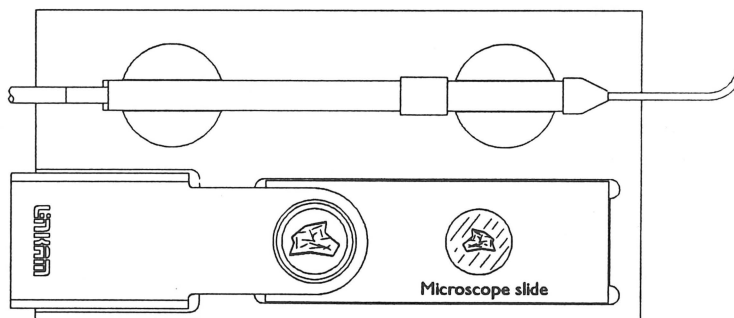
THMSG 600 SPARES

CRUCIBLE CARRIER	THMSG/CC2
STAINLESS STEEL CRUCIBLE	THMSG/CR
15 mm BY 0.3 mm SAPPHIRE WINDOW FOR THMSG/CR	WF15S
SET OF STAINLESS STEEL RINGS	RI17
16 mm BY 0.2 mm GLASS COVER SLIPS (100 OF)	W16G
2.4mm BY 1.6mm SAPPHIRE WINDOW FOR SILVER BLOCK	W600S
22mm BY 0.3 mm GLASS WINDOW FOR BOTTOM STAGE WINDOWS	W22G
7 mm BY 0.3 mm SAPPHIRE WINDOW FOR THMSG/SC	W7S
13 mm BY 0.2 mm GLASS COVER SLIPS (100 OF) FOR USE IN CRUCIBLE	W13G

Sample Loading Station

The sample loading station provides a clean and easy way to store the vacuum tweezers, crucible carrier and a microscope slide. Additional windows and samples may be kept on the station ready for loading into the carrier. Placing the window is also easier as the carrier is held in a flat position.

As the station holds the carrier flat, it makes the insertion and positioning of a new window using the vacuum tweezers much easier.



HFSG91 SAMPLE PREPARATION AND ACCESSORIES

When working with very small fluid inclusions both the optical performance and the temperature performance becomes critical. It is very important to avoid the use of too many glass cover slips as each layer effects the sharpness of the image seen by the microscope. The arrangement shown here uses only one glass cover slip over the silver lid and one in the top lid assembly as shown on the next page. However, if there is no condensation on the sample, the cover slip over the silver lid can be removed.

To obtain a very even temperature around the sample, a cover is placed over the silver block to form a miniature 'furnace'. With this arrangement the stage and programmer can achieve an accuracy of better than 0.2° C.

An alternative arrangement shown here uses a fixed window crucible which comprises of a stainless steel ring with a sapphire window fixed to the bottom. Because the crucible has a small lip around the top and will sit inside the crucible carrier without falling out, it can be used with the carrier to load a sample from the side door without removing the lid. The sample can be placed directly on the window or can be placed on a 7mm sapphire disc inside the crucible. For precision work the stainless steel rings and cover slips can be used to form the 'double glazed' environment.

When working with a synthetic fluid inclusion (e.g. CO₂) it should be placed directly on the crucible's window.

The crucible has the advantage over the silver lid assembly shown above, in that it is easier and quicker to use when looking at a large number of samples. Using the crucible does however, produce a very small error which can be calibrated out using the simple built-in sample calibration feature of the temperature programmer (see SAMPLE CALIBRATION FOR THE THM600/HFS91 STAGES in the programmer's reference manual).

Before changing the sample calibration of the programmer the following should be checked.

- 1) The silver block should be clean and flat so that the crucible 'sits' correctly on the block without any air gaps.
- 2) Make sure that the sapphire window in the silver block is level with the silver block top surface as this would cause the crucible to 'tilt' to a different position each time it is used and may cause the temperature values to be un-reproducible.

NOTE: Once the crucible arrangement has been used and if the programmer's sample calibration has been changed, the user must reset the sample calibration in the programmer before using the silver lid arrangement.

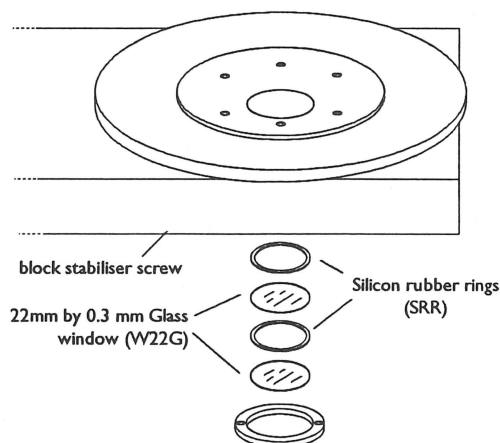
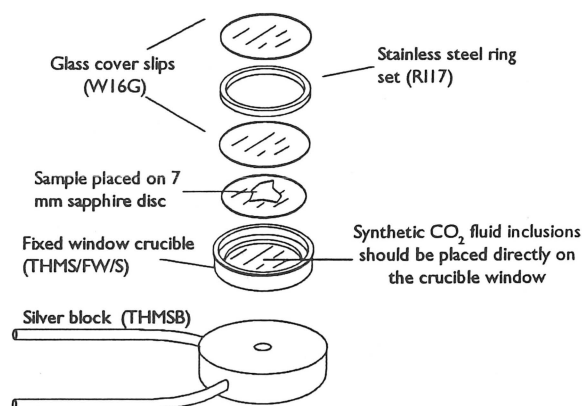
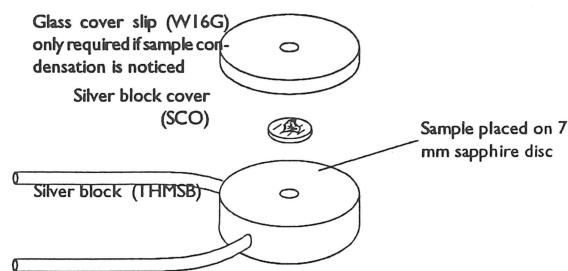
HFSG91 SPARES

FIXED WINDOW CRUCIBLE IN SAPPHIRE	THMS/FW/S
SPARE 15 mm BY 0.3 mm SAPPHIRE WINDOW FOR THMS/FW/S	W15S
SET OF STAINLESS STEEL RINGS	RI17
16 mm BY 0.2 mm GLASS COVER SLIPS (100 OF)	W16G
2.4mm BY 1.6mm SAPPHIRE WINDOW FOR SILVER BLOCK	W600S
22mm BY 0.3 mm GLASS WINDOW FOR BOTTOM STAGE WINDOWS	W22G
7 mm BY 0.3 mm SAPPHIRE WINDOW FOR THMSG/SC SAMPLE HOLDER	W7S
13 mm BY 0.2 mm GLASS COVER SLIPS (100 OF) FOR USE IN CRUCIBLE	W13G

THMSG600/HFSG91 BOTTOM WINDOW ASSEMBLY

If for some reason the bottom windows need to be replaced they should be re-assembled as shown in the diagram.

The block stabiliser screw is located on the bottom of the stage to the left of the window assembly as shown here.

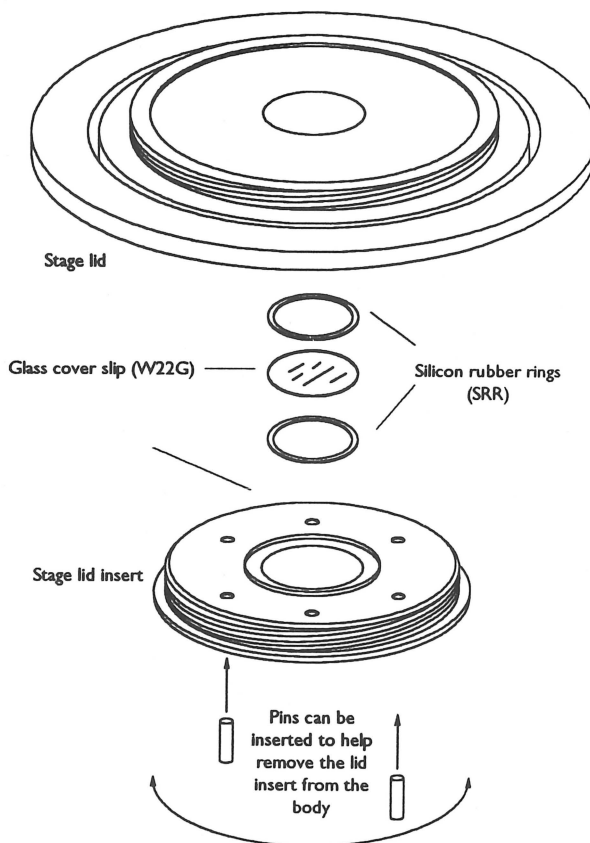


THMSG600/HFSG91 TOP LID ASSEMBLY

To replace the windows in the lid, the lid and insert should first be unscrewed from the body and taken apart. To un-screw the lid insert from the lid, place the two pins into any two holes in the lid insert and twist.

The stage lid insert should be turned upside down as shown and re-assembled in the order shown.

The stage lid should be screwed down until the cover slip is held firmly, then, turn the assembly over and screw down the insert until it is felt to come to a stop.



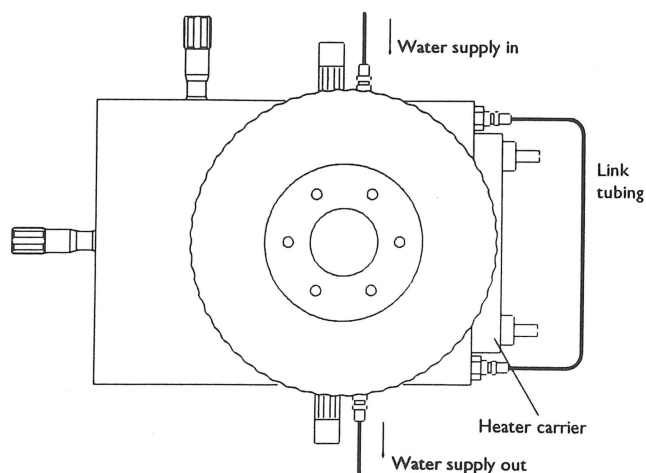
THMSG600/HFSG91 LID SPARES

22 mm BY 0.2 mm GLASS COVER SLIPS (100 OF) W22G
 22mm BY 0.5 mm QUARTZ WINDOW FOR
 BOTTOM STAGE WINDOWS W22Q
 SILICON RUBBER RINGS SRR

USING THE THMSG600/HFSG91 STAGE

A length of silicon tubing is supplied, which should be used for the top window, (i.e. to pass dry nitrogen through, which prevents condensation of the window at low temperatures) and for liquid nitrogen, when used to connect to the copper cooling coils of a Dewar flask.

Also supplied are four snap-on connectors for use with 3mm inside diameter, 6mm outside diameter flexible tubing. These are used for connecting both gas and water to the main body of the stage, although water cooling is only required if the stage is to be used at temperatures greater than 300°C and periods longer than fifteen minutes. The water supply should be connected as shown in the diagram. The HFSG91 should be connected in the same way i.e. a small length of tubing between the two connectors on the heater carrier.



THMSG600/HFSG91 SPARES

Platinum resistor 1.6 mm dia by 15 mm. 100 ohms PT100
 Set of O ring seals for the stage body and lid ORTHM
 Silver block heater assembly (exchange only) THMSB
 Silver block cover SCO

PROGRAMMING AT SUB-AMBIENT TEMPERATURES WITH A COPPER COIL AND DEWAR FLASK

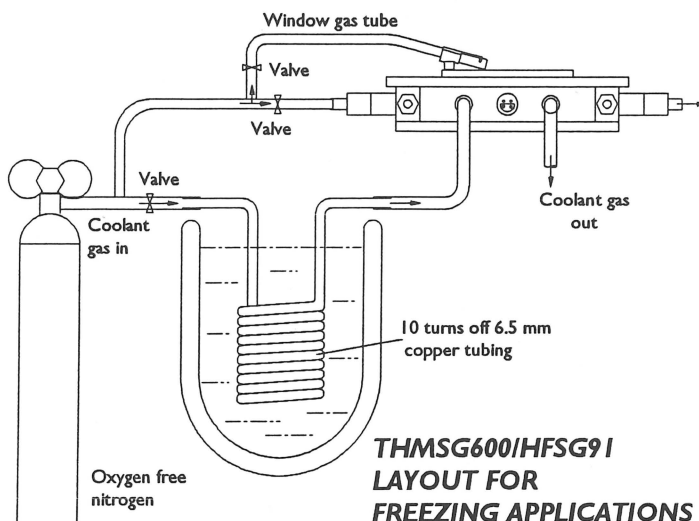
To utilise the full sub-ambient range of the Linkam programmers the coolant gas must be pre-chilled before entering the thermal block. This can be achieved most efficiently by passing oxygen-free nitrogen gas through a series of copper coils immersed in liquid nitrogen.

WARNING: IF COMPRESSED AIR IS USED, IT MUST BE COMPLETELY DE-OXYGENATED TO AVOID THE BUILD UP OF POTENTIALLY EXPLOSIVE LIQUID OXYGEN IN THE COOLING COILS

In normal use pre-chilled nitrogen can be used for temperatures down to -180°C .

If liquid nitrogen is pumped into the stage, temperatures as low as -196°C can be achieved in about 20 seconds. Because the action of the coolant is so effective, a much reduced flow of gas compared to that used for cooling at temperatures greater than ambient, will be required to obtain the desired cooling rate. If over-cooling occurs the "LOAD" light will remain permanently on, resulting in a loss of control. When heating from sub-ambient temperatures it is possible that the selected heating rate could be too slow for the flow of coolant gas resulting in the 'LOAD' led being permanently off, again resulting in a loss of control.

Therefore the flow of coolant gas must be made to balance the effect of the programmer's heating.



PROGRAMMING AT SUB-AMBIENT TEMPERATURES WITH THE CS196 COOLING UNIT

Description

The CS196 cooling pump can be used in conjunction with the THMSG600 or HSFG91 stage to cool a sample down to -196°C .

For applications where a small desktop dewar flask is more practical the 2 litre vessel with fitted pipe (D2L) can be used. Where longer experiments or where supply of liquid nitrogen makes it more convenient to use a larger vessel the DS1 dewar syphon can be used. Both devices work by drawing liquid nitrogen from a dewar flask through the cooling device via a filter, to prevent blockage from ice particles, where it vapourizes. The nitrogen gas is then passed into a valve which allows manual control of the flow rate. A small quantity of gas is also passed through the window gas tube to prevent condensation on the top surface of the lid window.

OPERATION

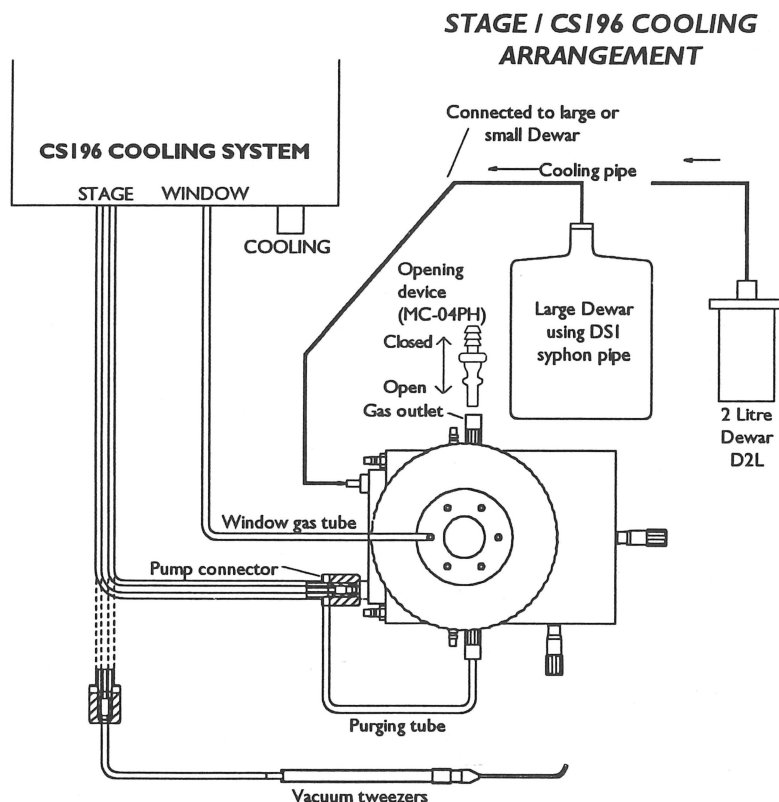
Filling the Dewar

When the dewar is filled, the lid should be placed on before the liquid nitrogen stops boiling. When this has finished clamp the lid into place. This is a precaution to prevent the water vapour in the air condensing in the liquid nitrogen.

Connecting the pump

Both the connectors from the dewar flask and the CS196 are simply pushed onto the cooling connections of the stage taking care to gently insert the capillary tube from the dewar.

A small tube from the outlet marked WINDOW on the CS196 should be connected to the top window gas tube as shown.



Operating the pump

When all connections have been made switch on the pump at the rear of the unit. The flow rate can be adjusted using the control marked "COOLING". Maximum flow is achieved at the -196°C setting and minimum flow at the 0°C setting.

Note: when holding at temperatures between these values the flow should be adjusted such that the programmers load light flashes only once or twice a second. This gives optimum control and efficient liquid nitrogen consumption.

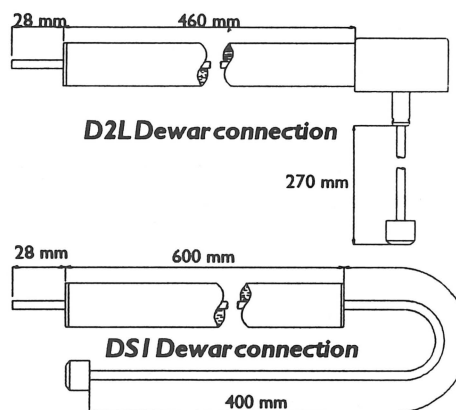
Precautions

If the lid is removed from the dewar flask ensure that the filter is at room temperature before it is re-inserted. This will prevent any ice blockage.

Do not draw air through the stage when it is below room temperature as problems with condensation and icing may occur.

Care must be taken not to damage or tightly bend the capillary tube as this will reduce the performance of the unit.

NOTE: UNDER NO CIRCUMSTANCES SHOULD THE TUBE LENGTHS BE CHANGED !



CS 196 FAULT DIAGNOSIS

Ensure that all the connections to the stage and dewar are correct as shown in the diagram and that the stage lid and top windows are properly sealed.

Problem:-

- 1) *The cooling rate is less than $30^{\circ}\text{C}/\text{minute}$.*

There can be several causes of this problem, the most likely being that one of the connectors has become blocked or damaged. Check that each tube is fitted tightly to the connector and that none of the tubing is twisted. This is particularly important on the stage connector as any constrictions of either the tubing or the connector will have a drastic effect on the cooling ability of the CS196. If the connectors and tubing are okay, check that the capillary tubing to the dewar flask is not bent or damaged and that the filter is intact and unblocked. If any damage has occurred to any of these items then it will be necessary to replace them. If no damage is found, check that the silver block is not constricted. This can be checked, simply by blowing through one of the coolant pipes.

- 2) *Stage will not cool to -196°C .*

Check that the stage lid is not touching the silver block when screwed down as this will cause a substantial loss of cooling ability.

- 3) *Condensation and ice forming on the window.*

Re-align the window gas tube clip to the required position in the stage lid.

COOLING PROCEDURE FOR STAGE AND CS196

If the THMSG 600 stage is to be used at low temperatures the stage clamps should always be used if possible. This prevents the stage from moving when the cooling pipe from the Dewar becomes less flexible due to the liquid nitrogen flow.

- 1) Connect the vacuum tweezers to the pump.
- 2) **THMSG 600 ONLY:** Open the end door of the stage and insert the sample holder (G7). Ensure that the small ring on the end of the sample carrier slides smoothly over the ramp and onto the silver block. The ring should lie as flat as possible on the block so that the sapphire disc will locate correctly. Adjust the manipulators so that the block window is in the centre of the ring.
- 3) **THMSG 600 ONLY:** Use the vacuum tweezers to place a 7mm sapphire disc inside the ring of the sample carrier.
HMSG 91 ONLY: Use the vacuum tweezers to place a 7mm sapphire disc onto the middle of the silver block.
- 4) Use the vacuum tweezers to place a sample onto the sapphire disc. Check that the sample does not touch the sample carrier ring as this can cause temperature errors.
- 5) Place the silver lid over the sample. For a THMSG 600 the two lines marked on the top of the lid, show the position of the side slot where the sample carrier moves.
- 6) Place a glass cover slip over the top of the silver lid which prevents condensation forming on the sample.
- 7) Screw the stage lid onto the body.
- 8) Turn off the pump and dis-connect the vacuum tweezers.
- 9) Connect the cooling pipe from the Dewar to the stage. Then, using a pushing and twisting movement, connect the CS196 pump connector to the stage. Connect the purging tube to the stage using the quick release gas connectors provided. (SEE STAGE / CS196 COOLING ARRANGEMENT DRAWING ON NEXT PAGE).

Open the gas outlet using the valve insert (MC-04PH). This should be pushed into the gas outlet which is on the opposite side of the stage.

Place the window gas tube into position on the top of the lid using the clip provided. The clip should be positioned in one of the six holes such that the lens turret of the microscope can be swung freely around. The end of the tube must be facing the centre of the stage lid window. Make sure that the lid is screwed down correctly onto the 'O' ring seals.

10) Switch on the CS196 cooling pump and set the control to -196°C.

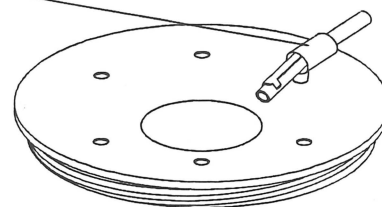
11) Switch on the programmer. Set the limit to 100°C and the rate to the fastest available. Press the START button and wait until the limit is reached.

12) Block the hole in the pump connector which is on the opposite side to the purging tube and block the window tube by pinching or folding it for about 30 seconds. This allows maximum flow of Nitrogen gas through the stage chamber to flush out most of the air. With the silver block at 100°C any remaining water vapour will condense on the colder tubes which carry the coolant to the block.

13) Remove both the valve insert (MC-04PH) and the purging tube. At about -150°C a partial vacuum will form inside the stage body which increases the cooling performance and prevents most of the condensation from occurring.

14) After this initial set up, the programmer can be set with the required limit and rate and used in the normal way. Adjust the CS196 control valve so that the load light on the programmer is flashing on and off evenly.

NOTE: If the stage is dismantled for any reason, special care should be taken to ensure that the 'O' ring seals are replaced correctly and to re-seal the block stabiliser screw. (see THMSG600/HFSG91 BOTTOM WINDOW ASSEMBLY)



USING THE STAGE WITH 80 AND 100X LENSES

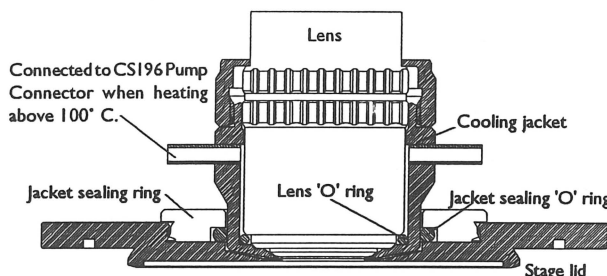
When using the stage with super long working distance lenses a specially designed lid and cooling jacket is required. The lens is placed inside a cooling jacket which is then inserted into the lid of the stage.

To increase the optical performance of the stage, the lid is sealed to the cooling jacket using 'O' rings, and does not therefore require a window. Once the chamber is purged, there should hardly be any condensation.

100 X Lens

To assemble the jacket and lid follow the steps below:

- 1) Mount the stage on the microscope's sub stage using the appropriate clamps. Using a 10X lens focused on the window of the silver block, adjust the stage position until the window is centred.
- 2) Adjust the condenser lens as described earlier.
- 3) Place the sample on the silver block.
- 4) Remove the lid from the stage body and check that the jacket sealing 'O' ring is in place.
- 5) Unscrew the two halves of the cooling jacket and make sure that the lens 'O' ring seal is in place. Unscrew the lens from the microscope and place it inside the jacket. Place the other half of the cooling jacket over the lens and screw the jacket together.
- 6) Tighten the jacket sealing ring until it just starts to pinch the 'O' ring. Push the cooling jacket assembly into the lid. It should be possible to pull out and push in to the lid smoothly and without too much force. If the jacket sealing ring is too tight the 'O' ring will bow and the seal between the jacket and lid will be lost.
- 7) Pull the assembly out of the lid and screw the lens into the microscope.
- 8) Screw the lid onto the stage. Gently move the stage up, until the cooling jacket is fully inside the jacket sealing ring. Adjust the focus.
- 9) Follow the cooling procedure outlined earlier.

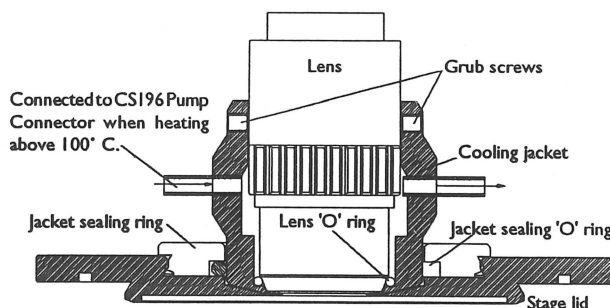


NOTE: Each time the cooling jacket is removed from the jacket sealing ring, or if a new sample is loaded the system will require re-purging.

80 X Lens

To assemble the jacket and lid follow the steps below:

- 1) Follow steps 1 to 4 as above.
- 2) Unscrew the lens from the microscope and place it inside the jacket until it comes to a stop. Gently tighten the grub screws to hold the lens in place.
- 3) Follow steps 6 to 9 as above.



PRECAUTIONS FOR USE WHEN HEATING ABOVE 100°C

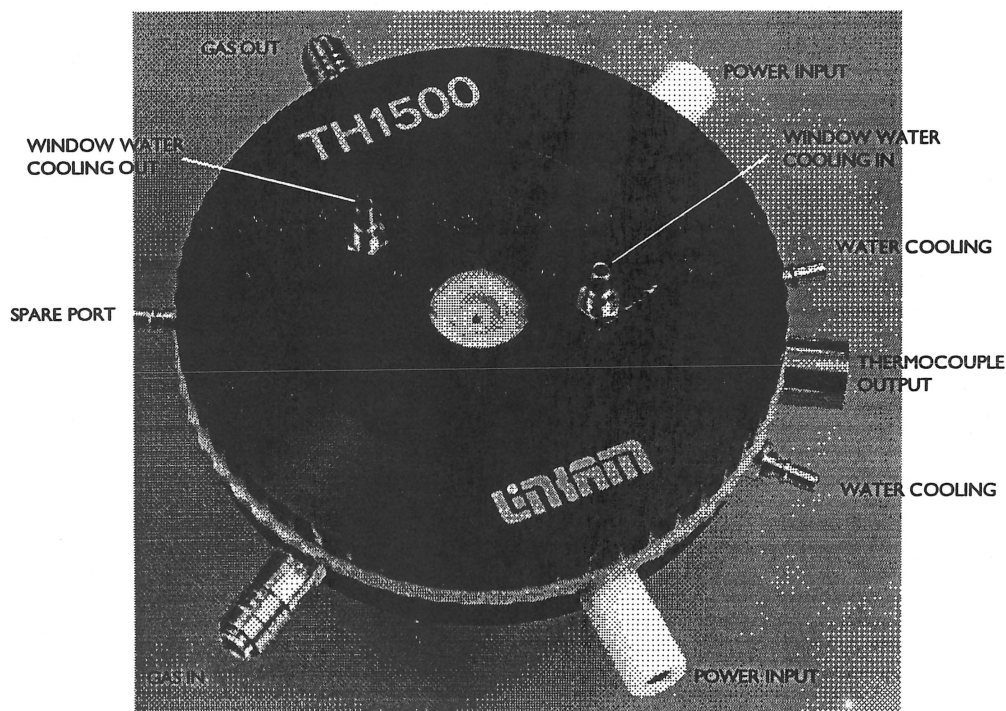
As the stage lid does not contain a window, the lens must be cooled using the cooling jacket and pump when heating the THMS stage above 100°C. This is achieved by removing the pump connector from the stage cooling pipes and connecting it instead to the pipe on the side of the cooling jacket.

THIS MUST BE DONE IN ORDER TO AVOID ANY DAMAGE TO THE LENS.

TH1500 STAGE

Installation

The stage should not be used above 400°C for any length of time without water cooling on both the body and the lid. Because the lid becomes hotter than the body the water should flow through the lid first and the body last. To make the connection between the body and the lid, a short length of tubing with snap-on connectors is supplied together with four other snap-on connectors for use with 3mm inside diameter, 6mm outside diameter flexible tubing. These are used for connecting both gas and water to the main body of the stage.



Using the TH1500 stage

To prolong the life of the stage, try to avoid consistently high heating rates and temperatures as this can lead to excessive grain growth in the platinum heater with the resulting effect that the wire becomes thinner and finally fails.

The TH1500 should not be switched off above 300°C as the sudden drop in temperature can cause the ceramic CUP and its supports to crack. A programmed cool to 300°C should always be used.

The TH1500 responds very quickly to changes in temperature on the sample, so to prevent any damage to the heater it is strongly advised that no COLD samples be introduced at higher temperatures and that the sample be as thin and as light as possible.

Connecting the stage to the power unit

The stage is connected to the PU1500 power unit via three wires. The two black wires supply power to the stage and simply plug into the rear of the power unit and into the two white PTFE connectors on the stage. The third wire which is the thermocouple signal from the stage, is connected to the power unit via the green cable and is plugged into the stage using a 4 pin metal bodied connector which is polarised in order to prevent reversal of the thermocouple output. If the connector is correctly aligned, no undue pressure should be required to insert the plug into the body of the hot stage.

Connecting the PU1500 to the TMS91

The power unit is supplied with a connecting cable comprising of a 9 way to 15 way 'D' type plug. Connect the 9 way 'D' type to the PU1500 and the 15 way 'D' type to the socket marked STAGE/PU1500 on the rear of the TMS91.

ALWAYS SWITCH THE POWER UNIT ON BEFORE THE TMS91

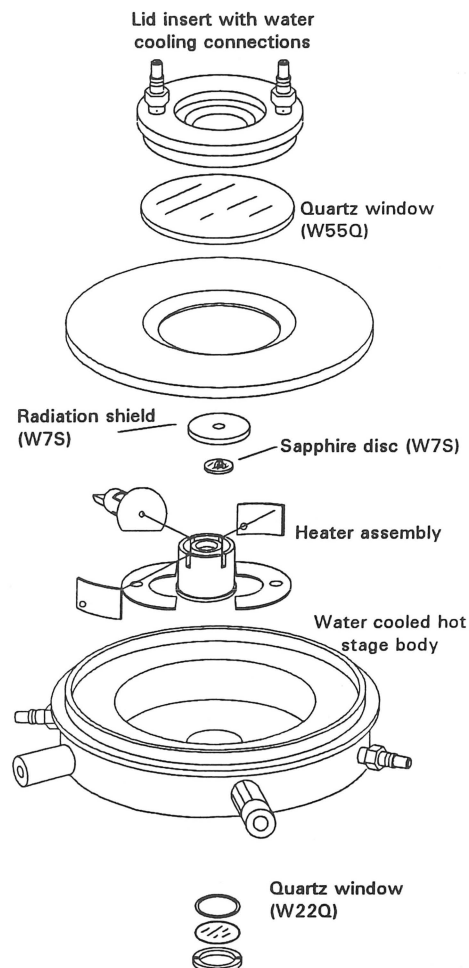
Stage heater protection

NOTE: The TH1500 stage is protected against overload and short circuit conditions by a 6.3 amp quick-blow fuse which is mounted in a fuseholder on the rear of the instrument.

If when replaced, the fuse breaks again, the heater winding resistance should be checked to see if a short circuit has occurred. The resistance of the winding when cold is approximately 0.4 ohms.

TH1500 HEATER AND LID ASSEMBLY

The diagram shows how the quartz windows are fitted into the TH1500 and if necessary how the element can be removed for repair.



TH1500 SPARES

SPARE ELEMENT FOR TH1500 STAGE	SE1500
RADIATION SHIELD	RS1500
SET OF O RING SEALS FOR STAGE BODY AND LID	ORTH1500
55 mm BY 2 mm QUARTZ WINDOW FOR TH1500 TOP LID	W55Q
22 mm BY 0.5 mm QUARTZ WINDOW FOR TH1500	
BOTTOM WINDOW	W22Q
7 mm BY 0.3 mm SAPPHIRE WINDOW FOR TH1500	
SAMPLE HOLDER	W7S

SAMPLE CALIBRATION SUBSTANCES

THE FOLLOWING SUBSTANCES AND THEIR MELTING POINTS ARE GIVEN AS A REFERENCE ONLY AND MAY BE DANGEROUS IF HANDLED INCORRECTLY

The melting point values for Benzoic acid and 2-Chloroanthraquinone are mean values taken from the dynamic and static methods used by the National Physical Laboratory.

SUBSTANCE	SYMBOL	T °C	MELT DESCRIPTION
Toluene	$C_6H_5CH_3$	-95	Simple digestion of needle by fluid which forms at the grain boundaries and within the needles.
Chloroform	$CHCl_3$	-63.5	Droplets of fluid appear within the crystals and progressively form a melt at the expense of a solid.
N-Octane	$CH_3(CH_2)_6CH_3$	-56.5	Droplets of liquid which can form internally and digest the small blades.
Chlorobenzene	C_6H_5Cl	-45	Droplets of liquid which form within the large blades and digest them.
Carbon Tetrachloride	CCl_4	-22.8	This coarsly crystalline aggregate changes to a featureless solid at -35 °C. Droplets of melt form within the solid and digest it.
N-Dodecane	$CH_3(CH_2)_{10}CH_3$	-9.6	A melt forms at the blade boundaries and the solid gas-bubble contacts.
Water	H_2O	0	Forms a coarsly crystalline aggregate on freezing. The melting occurs at the grain boundaries which appear more prominent before fusion.
Benzoic Acid	C_6H_5COOH	122	
2-Chloroanthraquinone	$C_{14}H_8O_2Cl$	210	
Potassium Nitrate	KNO_3	335	Phase change occurs at 127°C.
Potassium Dichromate	$K_2Cr_2O_7$	398	